MLF Experimental Report	提出日 Date of Report 2017/09/29
課題番号 Project No. 2017A0239 実験課題名 Title of experiment Powder Neutron Diffraction of Oxidation Products of Thermoelectric Materials 実験責任者名 Name of principal investigator Makio Kurisu 所属 Affiliation Ehime University	装置責任者 Name of responsible person Takashi Kamiyama 装置名 Name of Instrument/（BL No.） Super High Resolution Powder Diffractometer (BL-08) 実施日 Date of Experiment July 1-2, 2017

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。（適宜、図表添付のこと）
Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.

$(Sb_{1-x}Bi_x)_2Te_3$: raw and oxidized samples exposed at different temperatures.

2. 実験方法及び結果（実験がうまくいかなかった場合、その理由を記述してください。）
Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.

Experimental method

$(Sb_{1-x}Bi_x)_2Te_3$ compounds crystallizing in trigonal system were synthesized by melting constituent elements in vacuum. Then the ingots were pulverized to powders for oxidation at temperatures lower by 80 and 200 °C than their melting points. The powder neutron diffraction experiments for them were performed at room temperature by Super HRPD at BL-08. The refinement of crystal structures of raw compounds and their oxidized products were made for the obtained diffraction data (BS bank) by the Rietveld analysis using Z-Rietveld program.

Results

As one of the results, the neutron diffraction pattern of oxidized products of Sb_2Te_3 compound is shown in Figure 1. Oxidation of raw compound Sb_2Te_3 was made at 420 °C and for 13 hours. Refinement was made by assuming five products: Sb_2Te_3(raw), diantimony trioxides, diantimony tetraoxide and pure Te. The composition ratio and the lattice parameters are given in Table 1. Pure bulk tellurium is segregated by oxidation from the powder. Oxidation at higher temperature drives a transformation from diantimony trioxides (No. 227 and 56) to diantimony tetraoxides (No. 33 and 15).

2. 実験方法及び結果（つづき） Experimental method and results (continued)

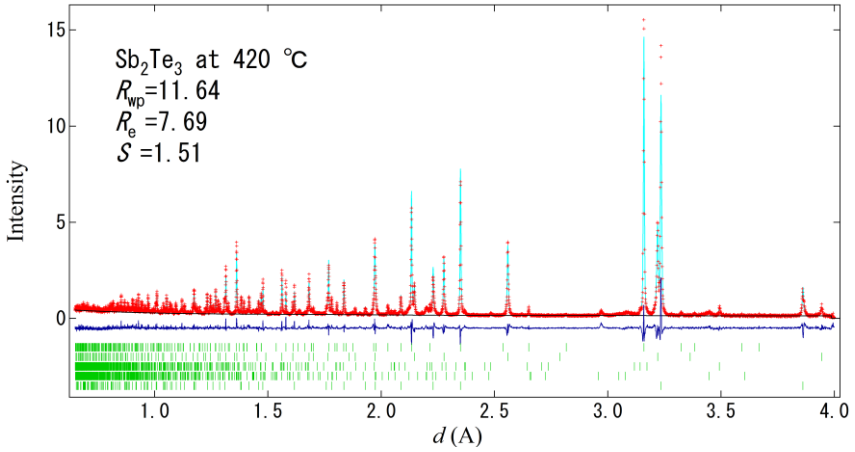

Figure 1. Neutron diffraction pattern of oxidation products of Sb_2Te_3.

Table 1. Composition ratio and lattice parameters of oxidation products of Sb_2Te_3.

Sb_2Te_3	oxidized at 420 ℃			
products	composition-ratio	*a* (Å)	*b* (Å)	*c* (Å)
Sb_2Te_3	0.1582	4.26834(3)	4.26835(3)	30.44976(79)
Sb_2O_3(227)	0.0336	11.15741(94)	11.15741(94)	11.15741(94)
Sb_2O_3(56)	0.0170	4.91337(105)	12.47129(285)	5.41486(118)
Sb_2O_4(33)	0.0502	5.45043(739)	4.80628(654)	11.83506(1632)
Te	0.7410	4.45882(8)	4.45882(8)	5.92413(19)